Semi-Annual Report

The MBE growth of InSb and InTlSb for Long-Wavelength Infrared Photodetectors and Focal Plane Arrays

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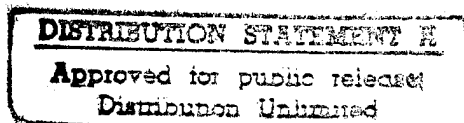ONR No. N00014-93-1-0409

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1. INTRODUCTION

There has been sustained interest in the area of narrow bandgap compound semiconductors due to their potential in many applications such as infrared photodetectors. These devices are essential for use in far infrared thermal imaging systems, pollution monitoring systems and free space communications systems. In fact, InSb large area detector arrays can be monolithically integrated into a single device along with the necessary signal processing components. InSb also has a low effective mass and narrow bandgap which results in its high electron mobility. This characteristic is useful in high frequency devices and magnetoresistive sensors for position sensing in the automotive industry. Current InSb photodetector technology uses bulk InSb material with ion implanted p and n layers. InSb substrates, however, are not semi-insulating which complicates monolithic integration and increases electrical noise. In addition, in order to increase the signal to noise ratio while decreasing substrate attenuation, InSb substrates require back lapping. Back lapping or substrate thinning causes material defects and increases fragility. In order to avoid these problems, epitaxial techniques such as MBE and MOCVD have been used to grow InSb films on GaAs and Si substrates. Recently, there has been interest in developing methods designed to more effectively accommodate the relatively large lattice mismatch between InSb and GaAs (14.4%) or Si (19.0%). In fact, much work has focused on improving material quality and reducing interfacial dislocations by incorporating AlSb buffer layers or thin Atomic Layer Epitaxy (ALE) InSb layers at the interface. AlSb, however, may not be suited for p-i-n structures due to its (i) indirect bandgap which reduces carrier mobility and device speed and (ii) poor conductivity. Although the ALE process produces improved carrier mobilities and X-ray rocking curve FWHMs this method has some drawbacks. For example, many shutter operations are involved which increases the probability of mechanical failure of the MBE system components and growth rates tend to be slow, ~0.1 μm/hr. Therefore, it is difficult to allow for efficient mass production of these devices with an ALE process. Hence, it is apparent that a reproducible, straightforward procedure is necessary to ensure the efficient growth of InSb p-i-n structures.

This report will cover the solid-source Molecular Beam Epitaxy (MBE) of various narrow gap semiconductor compounds focusing on InSb. The different surface reconstructions and surface phase transitions have been investigated using Reflection High Energy Electron Diffraction (RHEED). RHEED oscillations were observed for GaAs, InAs and InSb and have been used for determining growth rates and III/V flux ratio. The growth of InSb has proved particularly successful, InSb X-ray linewidths of 148 arcsec and mobilities of 92,300 cm^2/Vs at 77K for a 4.8 μm thick layer nucleated directly onto GaAs are the best reported to date. InSb p-i-n structures

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of 4.8 μm grown under the same conditions demonstrated a X-ray Full Width at Half Maximum of 101 arcsec and 131 arcsec for GaAs and Si substrates, respectively, and exhibited excellent uniformity of ±3 arcsec over a 3" substrate. Prototype InSb p-i-n detectors on Si have been fabricated and have demonstrated photovoltaic response at 6.4 μm up to 200K. These p-i-n detectors have also exhibited the highest D* for a device grown onto Si. A number of 2" and 3" wafers have been supplied to Loral Fairchild. These wafers have been fabricated into 256x256 focal plane arrays and have imaged when connected readout electronics, the first time for InSb grown on silicon.

2. BACKGROUND REVIEW

2.1 Material Properties of Bulk InSb

Indium Antimonide (InSb) is a direct, narrow gap semiconductor. It possesses one of the smallest energy gaps of all the direct gap semiconductors, which lends itself to device applications in the far infrared. InSb has a band gap of 0.17 eV at 300K and 0.23 eV at 77K corresponding to wavelength limits of 7 μm and 4.4 μm, respectively. InSb has a low effective mass and, as a consequence, an extremely high electron drift mobility compared to other III-V compounds (Table 1) making it potentially useful for high speed devices. Since InSb is a III-V semiconductor, well established processing and fabrication techniques can be used produce devices such as photodetectors. InSb has a lattice constant of 6.4788 Å there is a large lattice mismatch with both GaAs (~14.4%) and Si (~19.0%). This lattice mismatch can prove problematic during heteroepitaxial growth of InSb on GaAs or Si and it will be addressed in the following sections.

Table 1. Material properties at 300K of selected bulk III-V semiconductors.

	GaAs	InAs	InSb
Lattice Constant (Å)	4.6434	6.0484	6.4788
Energy Gap (eV)	1.43	0.36	0.17
Wavelength (μm)	0.87	3.44	7.0
Electron Effective Mass	0.067	0.028	0.013
Electron Mobility (cm^2/Vs)	8,400	22,600	100,000

Since the band gap of InSb is small, thermally exited carriers can drastically change the characteristics of the material. For example, the intrinsic carrier concentration has a strong dependence on temperature. A large carrier concentration leads to high dark current and noise in photodetectors. Therefore, a stable device operating temperature is critical for a good signal to noise ratio and reproducible results for these devices.

2.2 Principles of Molecular Beam Epitaxy

Molecular beam epitaxy (MBE) is a powerful technique for growing thin semiconductor layers of high purity. Thin films crystallize on a substrate surface, maintained at an elevated temperature in an ultrahigh vacuum, with the same crystalline structure of the underlying substrate. The composition of the grown layer depends on the constituent element beam fluxes or source evaporation rates. Since the growth rate of MBE is low, approximately 1 MLs^{-1}, surface migration of the chemiabsorbed elements can occur before they incorporate into the crystal. This allows for an extremely smooth surface and precise control of element composition which is necessary for lattice mismatch systems such as InSb on GaAs or Si. Since MBE growth occurs far from thermodynamic equilibrium, growth kinetics and reactions occur only at the outermost atomic layers of the substrate surface. Epitaxial growth by MBE involves a number of surface driven phenomena including: (i) the adsorption of constituent beam sources or elements onto the substrate surface, (ii) the dissociation and surface migration of adsorbed molecules and (iii) the thermal desorption of molecules ejected from the substrate surface. Since MBE is operated in an ultra-high vacuum (UHV) environment, it can be complemented with in-situ characterization techniques such as reflection high energy electron diffraction (RHEED) and Auger electron spectroscopy (AES).

Reflection High Energy Electron Diffraction
The primary strength of MBE is the ability to use Reflection High Energy Electron Diffraction (RHEED) as a characterization technique and monitoring system during epitaxial growth. An electron gun, typically 10-15 keV, is directed at a glancing angle (1-2°) on the substrate with the reflected or scattered electron beam viewed on a phosphor screen. RHEED provides both structural, and to a lessor extent chemical, information about the atomic arrangement of the structure of the crystal lattice and growth rates through RHEED oscillations. Since the electron beam is incident at a glancing angle, the penetration depth is normally only a few monolayers. Therefore, the information provided by the RHEED image only reflects the quality of the uppermost layers. For example, a smooth surface layer is usually represented by a streaky pattern, while a rough surface is represented by a spotty pattern. Hence, during optimum growth

conditions, layer by layer growth, a streaky pattern is visible. On the other hand, three dimensional island observed during nucleation of semiconductors with dissimilar lattice constants exhibits a spotty pattern is visible. In addition, a surface reconstruction, an additional periodicity at the surface, is normally observed corresponding to some minimum energy configuration of bulk lattice dangling bonds at the surface. The reconstruction depends on the specific substrate temperature and flux arrival rate. The most commonly observed surface reconstructions from III-V semiconductors are (4x4) and the (2x4) reconstructions. These reconstructions relate to different atomic surface coverage of various constituent elements (usually group V elements) and can provide information about growth conditions such as substrate temperature. RHEED can also be viewed in a temporal mode by utilizing the specular intensity oscillations that occur during growth. Maximum reflectivity occurs at a smooth annealed surface and when growth commences the surface periodically roughens due to the layer by layer growth mechanism. When the monolayer is half complete scattering is a maximum and the intensity is at a minimum. Therefore, as layer-by-layer growth proceeds, the specular spot in the RHEED pattern oscillates can be used to determine the growth rate.

2.3 Growth of InSb

The use of InSb for optoelectronic devices originated in 1961 with work by Guenther and Freller. These early films were grown using thermal evaporation techniques but hall coefficients were erratic and hall mobilities were lower than 20,000 $cm^2/Vsec$. The growth of InSb by evaporation techniques was not particularly successful. It was not until the development of advanced epitaxial techniques such as MBE and MOCVD that device quality InSb was grown, Table 2.

Table 2.   The properties of InSb on GaAs grown by MBE and MOCVD.

	MOCVD	MBE	MBE w\ALE
Thickness (μm)	3.6	4.0	4.0
X-ray (arcsec)	210	614	173
Electron Mobility (cm^2/Vsec)@77K	~80,000	46,400	104,000
Electron Mobility (cm^2/Vsec)@300K	~47,000	~40,000	~60,000
Background Concentration	n-type	n-type	n-type

MBE growth

Much of the recent MBE growth of InSb came from the Naval Research Laboratories (NRL). The NRL group have focused on trying to improve to the InSb/GaAs interface by minimizing the dislocation generated using low temperature growth and atomic layer epitaxy (ALE) buffer layers.

Thompson et al (1991) first deposited a low temperature (300 °C) growth of a 300 Å InSb interface layer prior to the main InSb active layer growth at 380 °C. Beam equivalent pressure measurements were used to control the growth rate of 0.1 μm/hr and 1 μm/hr for the interface and bulk layers respectively. X-ray data showed a FWHM of 240 arcsec and 140 arcsec for 2 μm and 4.4 μm thick samples, respectively. In addition, electron mobility results ranged from 28,800 cm^2/Vs and 44,100 cm^2/Vs at room temperature to 33,000 cm^2/Vs and 38,300 cm^2/Vs at 77K for the same 2 and 4.4 μm samples. The author attributed the decrease in mobility to defects present in the layer i.e. at lower temperatures, lattice scattering is reduced therefore impurity and defect scattering is enhanced.

Although the above results represented the best data at the time, the NRL group decided to incorporate an ALE buffer layer in order to enhance both the material's structural and electrical characteristics, Lee et al (1993). A typical structure was composed of an 84 atomic layer InSb buffer layer grown at a rate of 0.04 μm/hr at 300 °C and a 4 μm bulk InSb layer grown at a rate of 1 μm/hr. This work includes a comparison between various growth procedures including sample (A) with no buffer layer, sample (B) with a 300 Å low temperature layer and sample (C) with a 300 Å ALE layer. CTEM photographs show a large amount interfacial defects in sample A. The corresponding mobility at 77K and X-ray FWHM for sample A were 46,700 cm^2/Vs and 467 arcsec, respectively. Samples B and C exhibited 98,400 cm^2/Vs and 104,000 cm^2/Vs 77K mobilities with FWHM of 164 arcsec and 173 arcsec, respectively. The improvement in mobility for the samples with buffer layers was related to the obvious reduction in interfacial layer defects shown in the CTEM micrographs. A comprehensive study was conducted for InSb layers grown at various substrate temperatures and various ALE layer thicknesses. The optimum growth conditions were found to be a substrate temperature of 300 °C with a 40 monolayer InSb ALE growth followed by the bulk InSb layer at a substrate temperature of 420 °C. The best 4 μm sample had a X-ray FWHM of 124 arcsec, carrier concentrations in the low-10^{14}/cm^3 and 77K mobilities of 104,000 cm^2/Vs. Lee et al insisted that room temperature mobility measurements are not useful due to the fact that carrier concentration is dominated by intrinsic carrier concentration.

Another paper by NRL, Davis et al (1994), concerns the electrical characterization of one 14 μm InSb epilayer on GaAs. An initial InSb buffer layer with a thickness of 30 nm was grown using ALE at a growth of 0.1 μm/hr at 300 °C to reduce misfit interfacial dislocations. Hall measurements for this sample exhibited an electron mobility of 64,000 and 124,000 cm^2/Vs at 300K and 77K respectively. The corresponding X-ray FWHM was 41 arcsec. When the sample was etched down to 4 μm a 77K mobility of 110,000 cm^2/Vs and 70 arcsec for Xray FWHM were found. Data was obtained for thickness vs. FWHM, thickness vs. mobility and thickness vs. carrier concentration measurements.

Soderstrom et al (1992) completed the MBE growth of InSb directly on GaAs technique without the presence of an intermediate interfacial layer. A typical structure was composed of a 0.24 μm GaAs buffer layer grown at 600°C with InSb was then grown with a thickness of 2 μm at 400°C. The best room temperature mobility was 44,000 cm^2/Vs for a substrate temperature of 420°C. No 77K mobility data was given. The structural quality was examined using TEM. The author noted that only a few dislocations threaded through a thick 8 μm InSb layer. The reduction of the dislocation density with increasing InSb layer thickness was due to the reduction in dislocation interactions. This data represented the best reported for an InSb layer nucleated directly on GaAs by MBE.

MOCVD Growth

A number of groups have grown InSb by MOCVD. Recently Biefeld et al (1991) achieved room temperature mobilities of 61,000 cm2/Vs and 41000 cm2/Vs for 2.9 μm and 2.4 μm thick epilayers on GaAs, respectively. However, the corresponding FWHMs were 920 arcsec and 690 arcsec respectively. Nomarski microscopy revealed a set of irregular features with dimensions of approximately 0.4 μm x 0.4 μm which was attributed to the presence of excess indium during the growth of the initial InSb layer. A TEM study revealed the presence of misfit dislocations at the interface threading dislocations and stacking faults. A low temperature preliminary InSb layer to decrease dislocations was used similar to that used by the NRL group but modified to suit MOCVD growth. The low temperature layer acts as a trap for some of the threading dislocations and other defects as well as minimizing the surface roughness.

The MOCVD group from NRL, Gaskill et al (1991), have also reported the growth of high quality InSb. They reported a best FWHM, for a 4.4 μm InSb layer nucleated on GaAs, to be 190 arcsec with a corresponding 77K mobility of 90000 cm2/Vs. The group also reported results for InSb nucleated on a p-type InSb substrate. They achieved 64K mobilities of 2.43 x 10^4 and 2.2 x 10^4 cm2/Vs for a 4.8 μm sample.

Growth on Si

Until recently very poor results have been obtained for InSb grown onto Si. Most of these reported results have not shown equivalent InSb/Si material properties to those shown by InSb/GaAs. This could be due to the large lattice mismatch between Si and InSb, the high thermal-coefficient mismatch or the production of antiphase boundaries due to the polar/non-polar interface.

Chyi et al. (1988) presented some of the first results achieved for InSb grown on Si by MBE. They attempted to minimize the InSb-Si mismatch problem by preferentially introducing perfect edge dislocations by strained layer superlattices, using substrate tilting and in situ or ex situ annealing. Electron mobilities of 48,000 cm^2/Vs and 3,700 cm^2/Vs were measured at 300K and 77K with a X-ray FWHM of 474 arcsec for InSb nucleated onto a 0.3 μm GaAs buffer layer. However, when InSb was grown directly on Si the electron mobilities decreased to 39,000 cm^2/Vs and 700 cm^2/Vs while the FWHM improved slightly 410 arcsec.

Li et al. (1993) have found that the inclusion of an intermediate AlSb buffer layer during MBE growth appears to improve both electrical and structural characteristics by maintaining a wide range of temperatures (440-480°C) over which AlSb/InSb could be nucleated. In addition, it was hoped that lower lattice mismatch between AlSb and InSb, compared to that between InSb and GaAs, would improve the properties of the InSb epilayer. A basic structure was composed of a 3000 Å AlSb layer grown at 400 °C followed by a bulk InSb layer at 400°C. Typical results included electron mobilities of 44,000 cm^2/Vs for both GaAs and Si substrates. A structure composed of a 2 μm AlSb layer followed by a 3 μm InSb layer exhibited X-ray rocking curve FWHMs of 199 arcsec and 230 arcsec for GaAs and Si substrates respectively. According to the authors these values represent the best data reported at the time of publication.

Choi et al. (1993) have reported the first reasonable results for InSb grown on Si by MOCVD. They obtained a FWHM of 171 arcsec for a 3.1 μm InSb layer on GaAs. An InSb layer of 3 μm on GaAs/Si exhibited a FWHM of 361 arcsec and a room temperature mobility of 48000 cm^2/Vs. The higher FWHM was attributed to the higher dislocation density in the InSb layer. However, this was still the best reported value (at the time of publication) for InSb on Si, independent of growth technology. The Hall mobility vs. temperature plot showed anomalous results. For several samples of varying thicknesses, the mobility was maximum at approximately 240 K. The authors attributed the anomalous results to donor-like defects caused by the large mismatch and to a surface layer which dominates the transport in the material at low temperatures.

2.4 InSb p-i-n Detectors

In photodetectors, the objective is to absorb the photons and generate electron-hole pairs not larger than a diffusion length of a depletion region. The photogenerated carriers are swept out by the electric field, E, associated with the depletion region generating a current or voltage signal in the external circuitry. The width of the depletion region determines the transit time or response time of the device. A thinner depletion region will result in a faster device because it takes less time for the carriers to travel to the electrodes. However, the quantum efficiency of the detector decreases with a decrease in the depletion width. This phenomenon is due to the fact that a smaller number of photons are absorbed in a narrower depletion region. As a result, there is a loss of signal and sensitivity. A p-i-n structure is used to enhance the quantum efficiency of the device. Under reverse bias, the depletion width of the detector is the width of the intrinsic layer. The transit time in the intrinsic region, T, can be described by

$$T = \frac{d}{\mu E} = \frac{d^2}{\mu V_A}$$

where d is the width of the intrinsic region, V_A is the applied voltage, and μ is the electron mobility of InSb. InSb detectors possess several figures of merit. The two most important parameters are the Detectivity (D*) and the r_oA product. The D* value, for example, is defined as the inverse minimum power that a detector can see for a given bandwidth ($\Delta\lambda$) with a 1 cm^2 area and an amplifiers of a 1 Hz bandwidth. The detectivity is given by

$$D^* = \frac{(A\Delta f)^{1/2}}{P_s} \frac{V_s}{V_N}$$

where A is the effective detection area of each element, f is the bandwidth of the amplifier, P_S is the power at the detector, V_S is the signal voltage, and V_N is the noise voltage. The r_oA product on the other hand, is a good indication of degradation of resistance due to surface leakage or poor passivation. A typical r_oA value for InSb photodiode devices is approximately 2 x 10^6 ohm-cm^2. It is important to realize that as element sizes in the photodetector array (i.e. a focal plane array of InSb detectors) decreases, the r_oA product decreases as a result of increased surface leakage current. Therefore, the r_oA product is a useful value for the optimization of detector arrays.

Established InSb detector technology is primarily based on InSb substrates and this particular technology involves only bulk crystal growth. The doping required for the p-i-n structure are accomplished by ion implanting n and p layers into bulk InSb substrate material. In order to fabricate the device, multiple steps are necessary which can drastically decrease yield. For example, since the photodetectors are configured to operate with backside illumination (contacts and bonding on the front side), the bulk material must be thinned down to less than the minority carrier diffusion length. Without substrate thinning, the InSb substrate attenuates the signal due to photon absorption. The thinned structure is extremely brittle as a result of this thinning process and can contain many defects. In addition, for focal plane arrays the device array must then be attached to a silicon IC using indium bump bonding to provide the readout electronics. The ability to grow thin epitaxial layers of InSb directly onto Si would simplify this process.

3.   COMMISSIONING THE MBE SYSTEM

All samples were grown using an InteVac/EPI Model Modular Gen II solid source MBE reactor. This is the first MBE system that EPI delivered since they purchased InteVac. This system was unique because an alcohol chiller system was used to provide thermal isolation between the effusion cells as alternative to liquid nitrogen. When the system was installed a number of problems were resolved during the commissioning period. These include replacing the Pinnacle computer system that proved unreliable. In addition, the growth chamber ion pump gate valve had to replaced because the valve shaft failed after a few operations.

The new computer control system, Molly, works under a DOS operating system on a PC. But when the software was received, the control system was still in its development stage, many of the convenience and safety features of a standard control system were unavailable. We actively worked with EPI to tailor the system to our specifications. For example, communication links are being made between the control system and MBE components such as the ion gauges, RHEED, substrate rotation speed and orientation, and entry/exit heaters.

A number of new effusion cells were ordered and installed for the antimony and thallium sources. The use of antimony is particularly problematic because the vapor pressure is such that to provide sufficient flux for growth requires the effusion cell to be operated at temperatures close to the melting point of antimony. This normally results in the deposition of antimony around the lip of the cell or on the shutter that are slightly cooler that eventually make the cell inoperative. A modified dual filament Sb cell was used in a hot lip filament configuration to

prevent minimize the condensation of Sb. The Intevac-EPI system has four downward looking cells which means only four liquid sources can normally be used, in this case gallium, indium, aluminum and antimony. The thallium is also liquid at operating temperatures so a special inverted crucible was purchased to allow the downward looking configuration. The dopants were to be located in a downward looking location so they were premelted before being installed.

The source material inserted in the system includes Tl, Ga, In, Sb, Al and As source cells and Si and Be dopant cells. All the source material was high purity, 6 N's or better. All material was outgassed prior to any growth or calibration procedures. In order to optimize the MBE system for InSb-based material, indium and antimony are located in the lower positions. In these positions it is possible to fill the cells to maximum capacity.

During several of the InSb growths, large droplets of indium and gallium were observed on the surface of the sample. This was due to non-uniform heating of the source material. In addition, it was found that when the source cells were filled close to the recommended capacity material "spitting" would occur. The spitting generally happens when droplets of condensation form (due to radiative heat loss) at the crucible orifice and then roll back into the hot melt. The temperature difference between the liquid causes the ejection of small particles towards the substrate. These droplets degraded X-ray diffraction measurements and device quality (I-V characteristics and photovoltage response). As a result, the Intevac indium and gallium single filament cells were replaced with EPI 'hot lip' filament cells to improve the morphology of the samples. A thermal gradient along the length of the crucible that helps to minimize condensation and thereby eliminates spitting.

3.1 Outgassing/Baking

In order to preserve the operational integrity of the MBE system it is necessary to outgas and bake the MBE components. For example, a systematic process of outgassing is necessary when the source material is replenished; the sources cells are (a) purged of their remaining material by increasing the cell temperatures above their normal growth temperatures, (b) detached from the MBE system and filled with source material, and (c) attached to the MBE system and heated to remove any transient particles. After this process is completed, the entire system must be encased in "bake-out shrouds" which elevates the MBE system to 200 °C. This baking process desorbs and expels any water and other contaminants that may have entered into the system during the source replacement sequence. The system is normally baked for a minimum of 4 days in order to provide a clean, UHV environment within the main MBE chamber. The process of

outgassing is particularly important for narrow semiconductor materials since even a relatively small concentration of impurities can radically change the properties of the material produced. Steps must be taken to calibrate source fluxes for each of the new materials. Once the system bakeout was completed, each cell was individually outgassed and precalibrated using a Beam Monitoring Ion Gauge (BMIG).

3.2 Sample Preparation

The substrate quality and cleaning process is important since the grown epilayer quality depends critically upon them. Recently, the trend has been to use epi-ready material that is put directly into the growth system without any substrate preparation. This relies on the substrate manufacturer completing the correct etch to remove polish damage. This technology is only typically available for GaAs substrates. In most cases, the substrate cleaning procedure depends on the material type and corresponding etchant solution. The substrates are solvent degreased in boiling trichlorethlyene, acetone and methanol. The substrate is then rinsed in HDPI water. For GaAs, a H_2O_2/H_2SO_4 etch has been used. After etching, the substrate was thoroughly rinsed with HPDI water and blown dry with nitrogen.

A typical test sample is cleaved to about a 1 cm^2 piece. The sample is then bonded with indium onto a previously outgassed molybdenum block. The block is heated to approximately 200 °C with a hot plate and the melted indium is subsequently distributed evenly on the block. The substrate is worked onto the block by shifting the substrate in all directions. A good thermal contact results in an alloy between at the substrate and the moly block. It is extremely important to ensure a thin, even indium layer in order to achieve good thermal conductivity. A poor thermal contact, in most cases, results in poor surface morphology and uniformity.

All two and three inch wafers are mounted onto indium-free moly blocks with modified spring attachments. It is extremely important to ensure good spring tension to provide a good thermal contact. A poor thermal contact was apparent on our three inch wafers which exhibited a cloudy surface and poor surface homogeneity. As a result, the wafer holder was modified by including a 3" PBN plate which was inserted behind the wafer. The PBN plate, which has good thermal conductivity, improved the surface morphology.

The block is inserted into the entry/exit chamber which is pumped down to 10^{-7} torr. The chamber is then heated to 200 °C to remove water molecules and other impurities that may have entered into the system. The block is transferred into the buffer chamber where it is

subsequently outgassed for 1 hour at 240 °C to further remove any impurities from the surface of the substrate. The substrate is then transferred into the growth chamber in order to perform the oxide desorption.

The desorption process is conducted under an arsenic flux while the substrate temperature is slowly ramped up. At approximately 600 °C, the native oxide on the substrate desorbs, leaving the surface clean and ready for epitaxial growth. RHEED is used to monitor the thermal cleaning of the substrate and to determine the oxide desorption temperature. The oxide desorption temperature, T_{od}, and a clean substrate surface, can be identified by the temperature at which a clear, distinct streaky pattern is visible on the RHEED screen. System independent variables such as T_{od} provide a measure of the sample surface temperature independent of the thermocouple temperature. However, it is extremely important to identify T_{od} for each growth because the emissivity of a particular substrate and the substrate holder can vary depending on the recent history of the substrate holder.

InSb has been etched with a modified CP4A mixture however it is difficult to thermally desorb the oxide from InSb. The antimony oxide on the surface desorbs at a temperature of 515 °C close to the melting temperature of InSb at 527 °C. Alternative etches which result in a less thermally stable such as chloride based oxide are also being investigated.

3.3 Calibration Layers

Following the commissioning of a new MBE system, a series of calibration layers are grown to coat the interior of the growth chamber in order to minimize any changes in emissitivity during growth. Generally this is done by growing thick doped layers of GaAs with decreasing levels of doping concentration with each subsequent growth. Finally an undoped GaAs layer is grown that gives an indication of the operational characteristics of the system and the purity of the source materials. However, since the system installed in this laboratory is meant to primarily grow InSb based compounds, it made little sense to commission the system for GaAs based growth. So following the growth of few GaAs layers relatively thick InSb layers were grown to coat the system.

In order to calibrate each cell, several steps must be taken to obtain a good understanding of both the material flux rate and doping concentrations. Standard beam flux measurements are taken from the Beam Monitoring Ion Gauge (BMIG). However in most cases RHEED oscillation measurements are taken to provide an extremely accurate prediction of the growth rate of many

material systems. A typical RHEED oscillation has a specific intensity, phase and periodicity associated with each measurement. The periodicity can be used to provide an accurate measurement of the growth rate. In order to complete n- and p-type doping calibration, GaAs doped with silicon or beryllium are used to provide an accurate representation of the doping concentrations. InSb doping calibrations by capacitance-voltage measurements are not possible, therefore alternative methods such as Hall measurements must be used.

Calibration layers are primarily required for measuring the flux from the dopant cells since the RHEED oscillation method can not be used. In the process of commissioning the MBE system, a number of Si doped GaAs layers were grown that provided a rough measure of dopant concentrations from the dopant cell. The traditional method of calibrating the dopant cells is to CV (Capacitance-Voltage) profile a n-type or p-type doping staircases in GaAs. The dopant staircase is grown with a series of dopant steps of decreasing concentration and increasing thickness. Once the sample has been profiled, the dopant concentration can be normalized to a deposition rate of 1 MLs^{-1} for GaAs. The resulting Arrhenius plot gives the magnitude of the flux from dopant cell that can be easily corrected for changes in growth rate or lattice parameter when other material systems are grown. The apparent activation energy of sublimation for the dopant is measured for the sublimation of silicon or beryllium, Figure 1. In addition, it is necessary to recalibrate the dopant cells when the system has been opened to air.

Doping Staircases
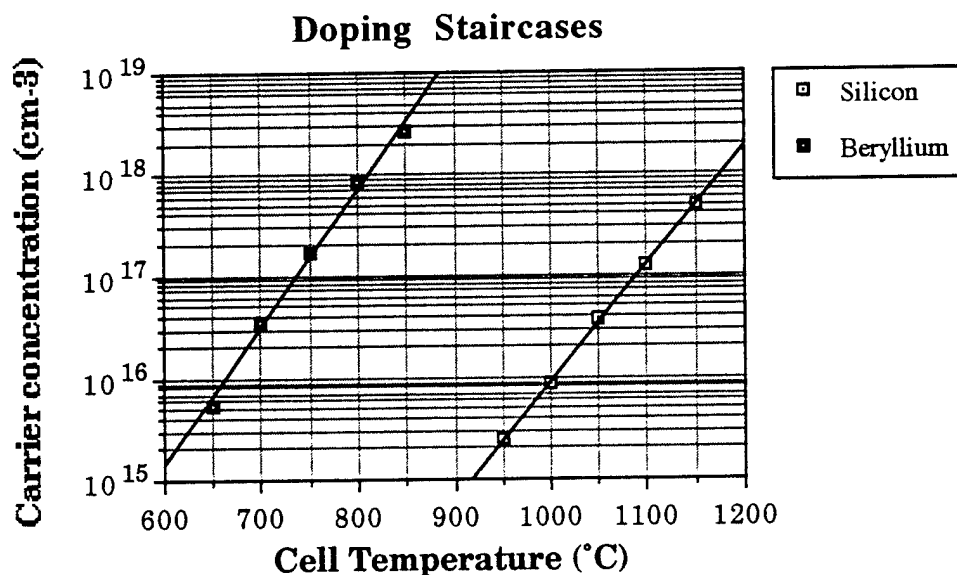
Figure 1. CV profile of a silicon and beryllium doped staircase in GaAs.

4. RESULTS AND DISCUSSION

4.1 Growth of undoped InAs

In order to check the purity of the individual source materials it is necessary to grow a whole matrix of different binary compounds such as GaAs, GaSb, InAs and InSb. For this reason InAs was grown under optimized growth conditions for this material system. The quality of the InAs critically depends on the growth temperature rather than the In/As flux ratio. High mobility InAs is normally grown at ~490°C, close to the (2x4) to (4x2) surface phase transition. Unfortunately this results in a layer with a rough morphology. The (4x2) reconstruction is due to an indium terminated surface and desorption of indium may be occurring during growth. A layer of thickness 3.4 μm was grown and this was confirmed by both etching and ball polishing measurements. A sharp intense peak was observed in the (004) reflection of the X-ray rocking curve of Full Width Half Maximum (FWHM) of 204 arcsec. The measured Hall mobility and carrier concentration of 1.2×10^4 cm^{2}V^{-1}s^{-1} and 9×10^{14} cm^{-3} at 300 K and 3.8×10^4 cm^{2}V^{-1}s^{-1} and 4×10^{14} cm^{-3} at 77K are comparable with the best reported elsewhere. But it should be stressed that it is difficult to do simple a comparison of Hall mobility due to the existence of a two-dimensional surface accumulation layer. These results are very promising considering that this was only the third layer to be grown after the MBE system was commissioned.

4.2 Surface phase transitions and RHEED Oscillations from InSb

Different surface reconstructions can be maintained over a large range of flux ratios and growth temperatures. Surface phase diagrams have proved to be a powerful tool in mapping out the surface reconstructions during both Langmuir evaporation and growth. This has helped in the identification of the optimum growth conditions for many different material systems. For example, the growth of high quality GaAs on a [100] substrate is normally achieved under an As stabilized (2x4) reconstruction at a growth temperature of 580 °C and a J_{As_4}/J_{Ga} flux ratio of greater than four. It should be stressed that there is no simple relationship between the material quality and surface reconstruction. But, the transitions between different surface reconstructions are clearly defined, especially during Langmuir evaporation. For example, the c(4x4) to (2x4) reconstruction is observed for an (001) GaAs surface at ~515 °C and is relatively insensitive to arsenic flux. This has can been used to provide another independent measure of the surface temperature during growth. For each substrate holder differences in the emissitivity result in the surface phase transitions occurring at different temperatures. Surface phase transitions have been used to provide a relative measure of the substrate temperature.

The use of surface transitions has proved particularly important for InSb because a transition between a c(4x4) to a(1x3) reconstruction on (001) InSb at ~390°C occurs almost at the optimum growth temperature for this material. The transition temperature depends on the antimony flux. In general, all growth temperatures are stated relative to a surface phase transition at a group V flux of I MLs^{-1}. If InSb is grown heteroepitaxially on GaAs the growth temperature is approximately 200°C from T_{od}. This results in a large error if the thermocouple is used to measure the temperature.

It has also been found to be relatively simple to freeze surface reconstructions on the (001) InSb surface that are normally only observed at high growth temperatures. The a(1x3) reconstruction is only observed above 390°C at normal antimony fluxes. On terminating growth, if the sample is turned away from the antimony source so that no antimony can accumulate on the surface, the a(1x3) reconstruction is observed even at room temperatures. This is important because in an attempt to relate the plethora of surface reconstructions observed in different material systems the concept of domain structures has been used to develop an universal model. Different coverage of the group V element (50-75 %) may explain the variation in spacing between the a(1x3) reconstruction on (001) InSb. Since this reconstruction can be easily obtained at room temperature it can be probed by Scanning Tunneling Microscopy.

RHEED oscillations can be used to measure the effective incorporation rates of both group III and group V species, to explore adatom surface migration lengths, and to probe interface quality and growth mechanisms. RHEED oscillations have been obtained during the growth of GaAs and InAs, subsequently nucleated onto a GaAs substrate. RHEED oscillations give an accurate measurement of the incorporation of the group III element which, is directly proportional to the flux incident on the surface if the sticking coefficient is unity. Oscillations have been observed over a period of minutes for the growth of GaAs with no evidence of beating due to flux uniformities.

RHEED oscillations provide a much more accurate method of calibrating InSb growth during MBE rather than using BMIG measurements. We have found that large errors can occur in BMIG measurements depending on the recent history of the ion gauge. RHEED oscillations provide a direct measurement of the incorporation of indium and antimony at the surface of the growing InSb. This is critically important for the growth of InSb because the vapor pressure of antimony is similar to that of indium. Growth cannot take place with a large excess group V flux

like GaAs because the additional antimony will accumulate on the surface and will either desorb or incorporate into the growing film.

An accurate measurement of the group III incorporation rate and thus absolute growth rate can be determined by measuring the variations in intensity of the specular spot in the RHEED pattern during growth. However, the observation of In-induced oscillations on InSb is not trivial because it requires the growth of an InSb buffer layer under optimized conditions. The physisorbed indium present on the surface appears to have a very large surface migration length. This means that in many instances it is easier to observe Sb-induced oscillations rather than In-induced oscillations particularly if the growth conditions are not properly optimized.

Since the sticking coefficient of the group V element is seldom unity and indeed is frequently zero in the absence of a group III flux a different procedure is used to calibrate group V element fluxes (dimers or tetramers) and incorporation rates. In the case of GaAs, a known excess of gallium is first deposited on the substrate surface, with no arsenic flux present. This leads to the formation of isolated islands of gallium. Intensity oscillations are then induced by the arsenic flux and they continue until all of the surface excess of gallium has been consumed. Using the same technique to calibrate group V incorporation in InSb, Sb_4-induced RHEED oscillations were observed following the controlled deposition of ~10 monolayers of indium onto a (001) InSb epilayer that had been nucleated onto a GaAs substrate. The period of the oscillation gives an accurate incorporation rate for antimony on InSb, Figure 2.
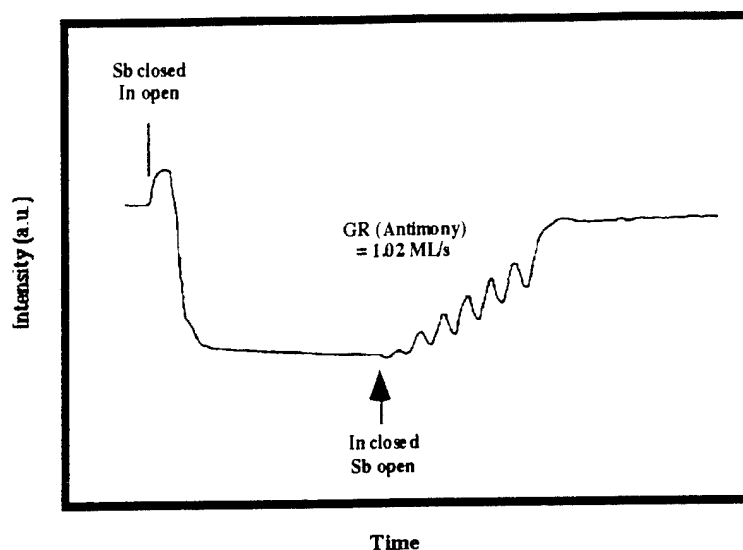
Figure 2. Sb-induced RHEED intensity oscillations. An excess of In is accumulated on the InSb surface and oscillations are observed until this excess is depleted.

A systematic calibration of Sb_4 incorporation was completed as a function of the antimony cell temperature at a constant substrate temperature of T_t-30 °C and is shown in the form of an Arrhenius plot in Figure 3.
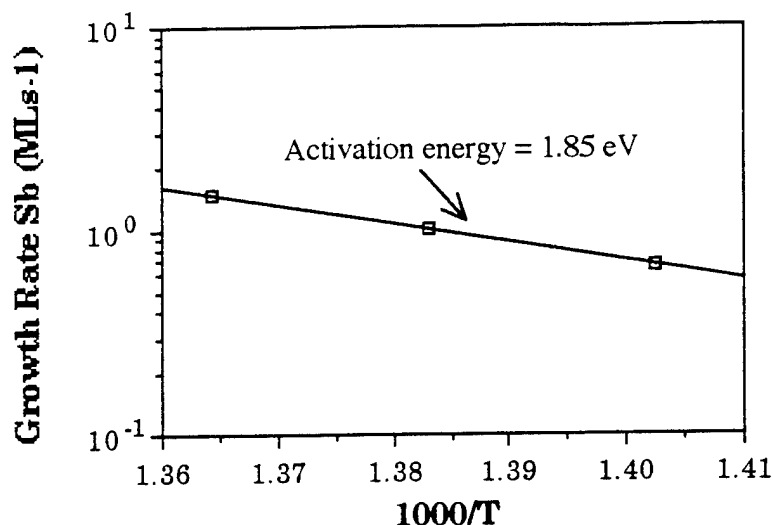
Figure 3. An Arrhenius plot of the apparent activation energy of Sb_4 sublimation for a hot lipped effusion cell, 1.84 eV (solid line), obtained from Sb-induced oscillations.

The apparent activation energy of sublimation for Sb_4 was $E_{Sb}=1.84$ eV. This is close to the activation energy for the sublimation of antimony over antimony, 1.38 eV, suggesting the vapor pressure of antimony from the effusion cell has been measured. The slight difference in the measured and theoretical value is due to the construction of the hot lipped cell. The hot lipped cell is heated at the lip, as the name implies, so the thermocouple at the rear of the cell only measures a part of some average value. It is interesting to note that there is some evidence that the sticking coefficient of antimony is unity even when Sb_4 is used and this will be investigated further.

4.3 Growth of high quality undoped InSb

A new MBE system was commissioned specifically to grow InSb based compounds. At first, it is necessary to show the ability of the MBE system to grow high quality InSb layers. Hence, a series of undoped InSb layers have been grown to optimize the structural, electrical and optical properties of this material. The system independent parameter of T_t and the observation of RHEED oscillations have been used to calibrate the growth conditions. To date, InSb has been grown at temperatures around T_t and In/Sb flux ratios varying between 1.25 to 1.88 These growth conditions are known to produce the best quality material. All the InSb layers have been

grown heteroepitaxially onto semi-insulating GaAs substrates. The epilayers have been grown with the same nominal thickness of 3.3 μm because both the structural and electrical properties depend strongly on thickness. The FWHM of the x-ray peaks have a strong correlation with the number of dislocations. Therefore, as the layer thickness increases the FWHM decreases. The mobility measurements also depend on thickness because one or possibly two channels of parallel conduction exist that are independent of layer thickness. Therefore, as the layer thickness increases, the mobility increases. The important data obtained to date is summarized in Table # below.

Table 3. Undoped 3.3 μm InSb epilayers nucleated directly on GaAs

Sample #	Temperature ($T_t = 390°C$)	Flux ratio (In/Sb)	X-ray (arcsec)	μ at 300K ($cm^2V^{-1}s^{-1}$)	n at 300K (cm^{-3})	μ at 77K ($cm^2V^{-1}s^{-1}$)	n at 77K (cm^{-3})
ssmbe004	$T_t + 5°C$	0.8/1.5	292	21600	3.6×10^{17}	30600	1.1×10^{17}
ssmbe008	$T_t + 30°C$	0.8/1.5	183	40400	4.9×10^{16}	34000	4.9×10^{16}
ssmbe010	$T_t + 5°C$	0.8/1.0	193	59400	2.7×10^{16}	81400	1.7×10^{16}

In Table 3, the layer grown at a temperature of $T_t + 5$ °C (394 °C) and a III/V incorporation ratio of 0.8/1.0 exhibited the best overall properties showing an X-ray FWHM of 193 arcsec, with 300K and 77 K mobilities of 59,400 and 81,400 $cm^2V^{-1}s^{-1}$, respectively. All the samples in Table I had a clearly defined absorption edge at 5.5 μm in the photoconductivity spectra, independent of the X-ray and electrical measurements. The results summarized in Table I are the best reported to date for InSb nucleated directed onto GaAs.

In previous work it was thought that the interface between the InSb and GaAs, which was highly dislocated due to the lattice mismatch, would dominate the physical properties of the layer. Therefore, two approaches were used to minimize the effects of this region by either growing a low temperature ALE buffer layer at 300 °C or growing an AlSb buffer layer. In both instances, the quality of the material grown did not exceed the results reported here. In addition, both techniques have drawbacks, such as the poor conductivity of AlSb, and the increased probability of mechanical failure associated with the ALE process. It is suggested that carefully controlling the growth parameters during the nucleation process is critical for producing high quality material. The quality of material reported here is superior to that obtained by other groups when they did not use ALE or AlSb buffer layers.

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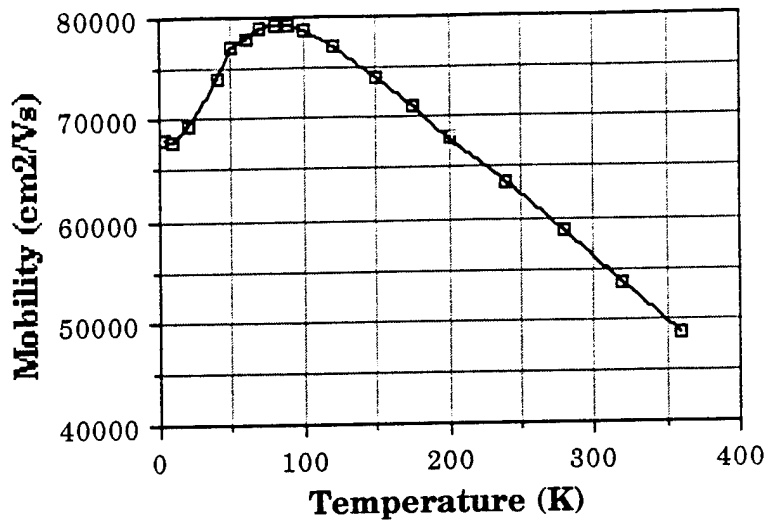
Figure 4. Temperature Hall mobility of InSb for 3.3 μm thick layer nucleated directly onto GaAs, sample SSMBE010.

Figure 4 shows the mobility versus temperature for InSb layer grown under optimum conditions. The characteristic temperature dependence of a peak in the mobility around 80K indicates that the InSb epilayer has a bulk-like n-type background. Ionized impurity scattering limits the mobility at low temperatures 4-77K whereas optical phonon scattering dominates from 77-300K. Dislocation scattering is also important but, as will be seen, this depends strongly on layer thickness. The observation of n-type behavior is in contrast to much of the work that had been reported previously which describes InSb epilayers with p-type characteristics. In this instance, the peak in mobility normally occurs around 240K, a Hall coefficient sign reversal is observed and Hall mobility at 77K is typically 20,000 $cm^2V^{-1}s^{-1}$.

To further investigate the thickness dependence of the Hall mobility, a 4.8 μm layer was grown under the optimum conditions described above. The X-ray FWHM for this layer was 148 arcsec (Figure 5) and the 300K and 77K Hall mobilities were measured to be 62,300 and 92,300 $cm^2V^{-1}s^{-1}$, respectively. This a large improvement to those results previously reported Thompson et al (1991) who obtained a X-ray FWHM of 614 arcsec and a 77K Hall mobilities of 46700 $cm^2V^{-1}s^{-1}$ for a 4 μm InSb layer grown directly on GaAs.

SSMBE026 : InSb on GaAs
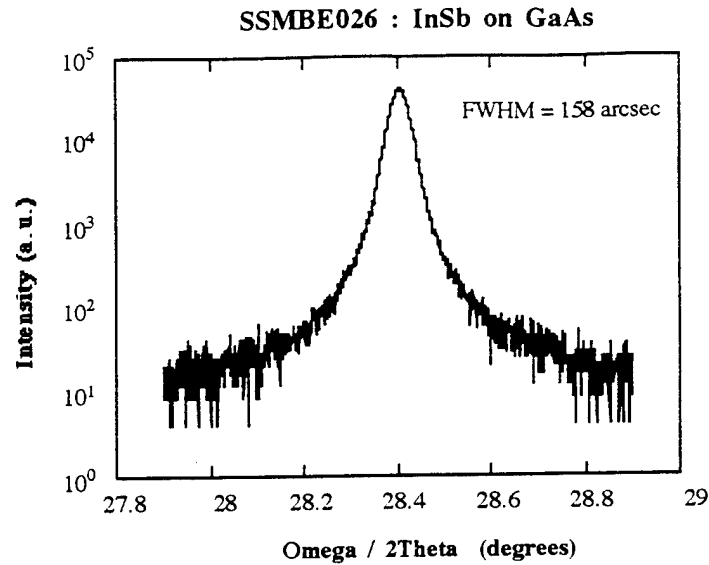
Figure 5.   The (004) reflection of the X-ray rocking curve of an InSb layer nucleated directly onto GaAs.

The 4.8 μm sample was used for step-and-etch measurements. The InSb layer was etched with a lactic/nitric acid solution to reduce the thickness which was followed by a Hall measurement. The 300K and 77K mobility versus thickness dependence is shown in Figure 6. The step-and-etch measurement also shows the reproducibility of the growth process; the 77K mobility of the 3.3 μm sample in Figure 4 is the same as that for the 3.3 μm thickness in Figure 4 ($\approx$ 80,000 $cm^2V^{-1}s^{-1}$).

InSb Hall Mobility vs Thickness
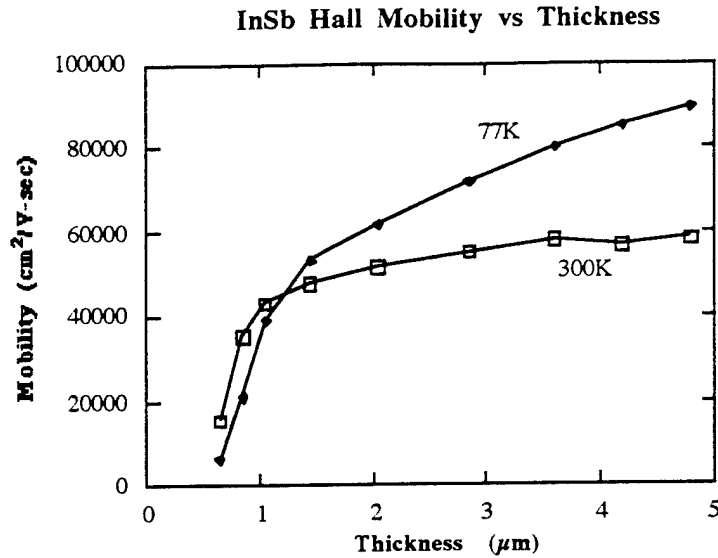
Figure 6. Mobility versus thickness for a 4.8 μm InSb epilayer grown under RHEED optimized conditions.

The electrical characteristics of the InSb epilayer in Figures 4 and 6 can be understood by considering the epilayer to be composed of three layers: a surface layer due to carrier accumulation or inversion, a bulk-like layer with reduced or negligible dislocation density, and a highly dislocated interface layer.

The 77K Hall mobility is primarily affected by a surface electron accumulation or inversion layer in the thickness range from 4.8 to 2 μm. The mobility of electrons at the surface is lower than that in the bulk-like layer because additional scattering mechanisms exist. If the electron density at the surface is large enough, the surface layer may be conductive enough to affect the low temperature measurements on unintentionally doped samples. This is less likely at higher temperatures (300K) since the intrinsic electron concentration will be higher which makes it more conductive compared to the low temperature case. At 77K, the mobility decreases with decreasing thickness because of the relative increase in the thickness of the surface layer compared to the bulk-like layer. At 300K, the mobility is almost constant over the same thickness range because mobility is less affected by the surface layer due to the increased carrier concentration of the bulk-like layer at this temperature.

As the layer thickness decreases, the effect of the surface layer will continue to increase. However, it is apparent that another scattering mechanism begins to dominate the mobility and this equally affects the 300K and 77K mobility. The interface layer has a negligible effect on the Hall measurements as long as the total epilayer thickness is larger than the thickness of the highly dislocated layer at the interface. Below some critical value, dislocation scattering becomes dominant and results in a decrease in mobility around $1 \mu m$. Due to this mechanism, the mobility approaches zero before zero thickness.

Doping InSb

Some silicon and beryllium doped layers of InSb have also been grown in preparation for p-i-n photodiode structures. These are generally the best behaved and most commonly used shallow acceptor and donor dopants in MBE growth. The silicon behavior is complicated because it is a group IV element, and is amphoteric in nature. This is particularly true at the high doping levels and high growth temperatures, $T_t + 30$ °C. The highest doping levels reported for InSb are 3-4 x $10^{18} cm^{-3}$ for growth temperatures of $\sim T_t$-40° C, however; if the growth temperature is too low the silicon is not electrically active. For doping levels of $<10^{18} cm^{-3}$ silicon is non-amphoteric favoring the indium site if the growth temperature is kept below T_t. InSb has been grown with a silicon doping concentration of 2 x $10^{18} cm^{-3}$ at a growth temperature of T_t-30 °C. No attempt has been made to optimize the growth for higher doping concentrations at this time. More recently however, the silicon was replaced by a mono GaTe source allowing n-type doping concentrations in the low $10^{19} cm^{-3}$. The beryllium permits controllable doping levels between 10^{14} to $10^{19} cm^3$ with 100% electrically active centers. InSb has also been grown with a beryllium doping concentration of 2 x $10^{18} cm^{-3}$ at a growth temperature of T_t-30 °C.

4.4 InSb Photodetectors

The primary interest in the epitaxial growth of InSb on GaAs and Si for has been for photodetector applications. Current InSb photodetector technology typically uses bulk InSb substrates with ion implanted p- and n- layers. Semi-insulating InSb substrates cannot be produced which complicates monolithic integration and increases electrical noise in discrete devices. In order to increase the signal to noise ratio while decreasing substrate attenuation, InSb arrays require back lapping when operated under backside illumination, the normal mode of operation. InSb photodetectors are important for far infrared thermal imaging however their use is severely limited by the difficulty of producing large area focal plane arrays. For example, the indium bump technique for mating the active device in an array with its control electronics is prone to failure and gives limited success despite, in some instances, a large investment of time

and resources. A major step forward would occur if the technology of producing InSb large area detector arrays could be simplified so that the signal processing components are monolithically integrated into each element, particularly during growth. An important part of developing this type of technology has been the ability to grow high quality InSb films on Si and GaAs, described above. This section reports fabrication and characterisation of prototype InSb p-i-n photodetectors grown on GaAs and GaAs coated Si. These devices have shown good operational characteristics with a photovoltaic response up to 6.4 μm at 200K and a $D_{BB}^*(800^\circ C)$ of 7×10^8 cmHz$^{1/2}$W^{-1} at 77K even though these devices have not been optimized.

Several different InSb p-i-n photodetectors have been grown on GaAs coated Si substrates for operation at 77K. A typical structure is shown in Figure 7. A thin GaAs buffer layer of ~300A is first grown to improve the sample surface prior to nucleating the InSb. The n-type layer is grown >1μm in order to decrease dislocation scattering at the interface, and doped at 4×10^{16} cm^{-3} to minimize free carrier absorption. A thick 4 μm undoped intrinsic region is then grown to maximize the signal to noise ratio and optical gain. Low intrinsic region carrier concentrations are desired to decrease dark current and noise. Recently, by changing the antimony source material to 7N the carrier concentration has decreased by approximately an order of magnitude at 77K from 1.1×10^{16} cm^{-3} to 2.2×10^{15} cm^{-3}. A thin, typically 0.5 μm, highly doped p-type region necessary for a small, localized depletion region and good conductivity is then grown. Mesa structures with dimensions of 400 μm x 400 μm were fabricated using standard photolithographic techniques and a lactic/nitric acid etchant. The ohmic contact for both n- and p-type materials was a 40 nm Ti layer followed by a 300 nm Au layer; both layers were deposited using electron beam evaporation. The contact patterns, which were 150 μm x 150 μm, were made by using photolithographic lift-off and selective chemical etching techniques. The assembly process, including die separation, mounting, and wire bonding were again accomplished using standard fabrication techniques.

Ti/Au

| P+-InSb |
| undoped InSb |

| N+-InSb | Ti/Au |

n-GaAs, GaAs, GaAs-coated Si substrates
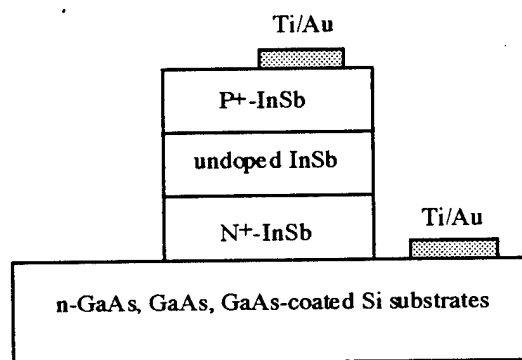
Figure 7.   Typical p-i-n photodetector device structure

These prototype devices structures showed excellent X-ray and photovoltage properties. A FWHM of 101 arcsec was obtained for a 4.8 μm thick InSb p-i-n structure on GaAs. In addition, a FWHM of 131 arcsec was measured from a 4.8 μm thick InSb p-i-n structure on Si. This is by far the best structural properties for InSb grown onto these substrate materials. Both these p-i-n structures were grown directly onto 3" substrates. X-ray mapping techniques and Nomarski microscopy revealed excellent crystalline and morphological uniformity. In fact, the X-ray FWHM uniformity was better than ± 3 arcsec over the entire surface of the 3" wafer. The photovoltage response for the InSb detector on Si was measured using a Mattson Fourier Transform Infrared Spectrometer and is shown in the Figure 8. The data shows a sharp, band edge with a photovoltaic response at 6.4 μm up to 200 K due to the excellent material quality. The absolute photoresponse was obtained using a Mikron 304 calibrated source and standard lock-in detection techniques.
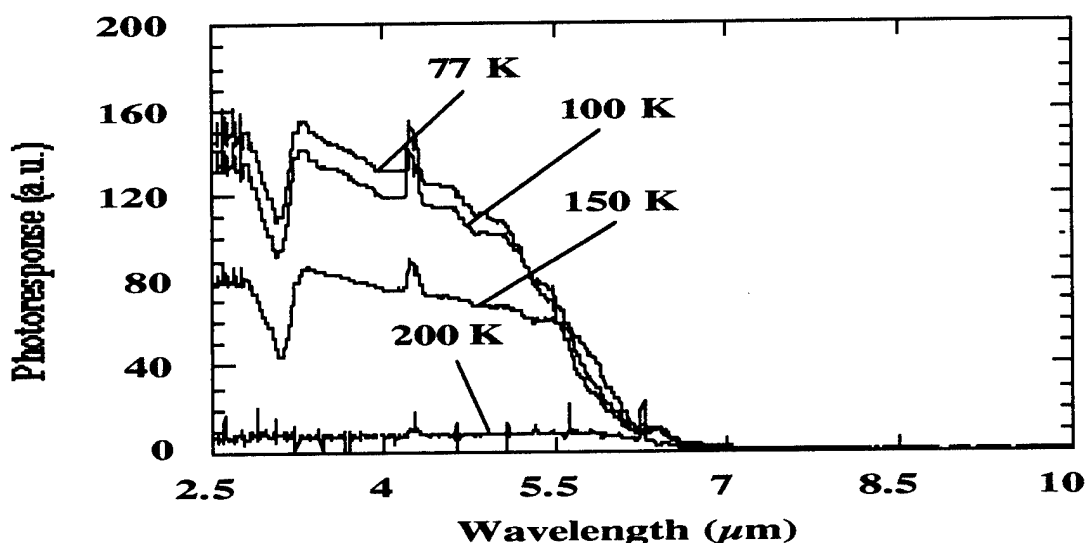
Figure 8.   Photovoltage response of an InSb p-i-n detector on Si

The temperature dependent D_{BB}^* at 800°C is shown in Figure 9. $D_{BB}^*(800°C)$ is 7×10^8 cmHz$^{1/2}$W^{-1} at 77K and is almost constant at this value up to 150 K. The peak value of D^* should be about an order of magnitude higher which is good considering this is for an unoptimised structure grown on Si. Above 150 K, $D_{BB}^*(800°C)$ drops with a corresponding

reduction in the photoresponse. These characteristics represent the best results reported in the literature for an InSb detector structure grown on Si. However, much work still has to be completed to grow and optimize a device that can operate with similar parameters to bulk InSb.
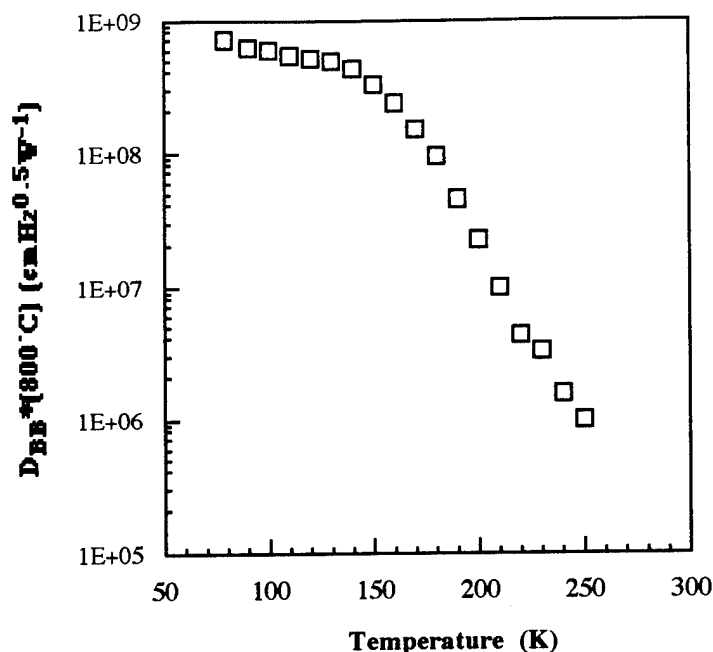
Figure 9. Temperature dependent $D_{BB}*(800°C)$ of the p-i-n photodetector shown in Figure 8

A number of p-i-n structures grown on 2" (five samples) and 3" (eight samples) silicon wafers have been sent to Loral Fairchild Corporation for fabrication into focal plane arrays, Appendix 8.1. The wafers have been processed into a 256 x 256 array and bump mounted onto readout electronics. These focal plane arrays have successfully imaged through the Si substrate material. This is the first time images have been obtained for InSb grown on Si reflecting the high quality of the InSb growth, Appendix 8.2.

4.4 Growth of InTlSb

Growth of InTlSb has been investigated. Thallium incorporation into InSb is not a simple task. There has only been one report of the growth of InTlSb by MBE in that work Tl was only incorporated at doping levels. A number of InTlSb layers have been grown by MBE to investigate the incorporation of Tl into InSb. Early samples showed the existence of islands on the surface. So preliminary characterization of the InTlSb layer was completed using Scanning

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Electron Microscopy and EDAX (Energy Dispersive Analysis of X-rays) measurements to understand the composition of these islands. It was found from SEM data that the observed thallium composition is dependent on the substrate temperature (Figure 10).
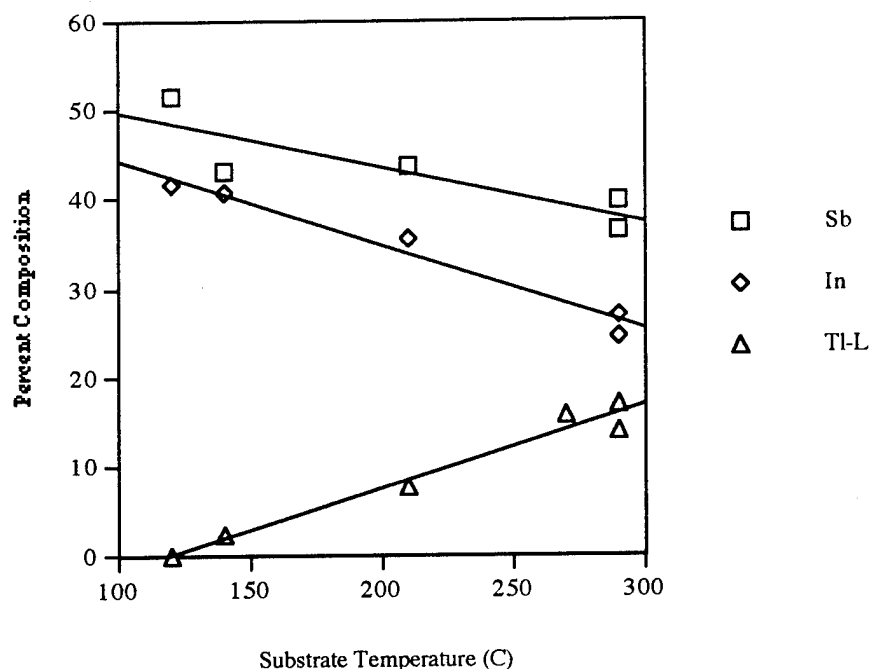
Figure 10.   EDAX Composition Analysis of In(Tl)Sb layers.

There is a very good correlation between the thallium percent composition and the surface density of the islands. It appears that an increase in substrate temperature results in an increase in the surface area, density ratio and thallium percent composition of the islands. As expected, an increase in thallium composition (and island density) results in a decrease in indium composition (since we theoretically have $In_{1-x}Tl_xSb$). In addition, a corresponding increase in thallium results in a decrease in antimony composition. This result suggests two possibilities.

1)   The thallium is replacing indium on the surface of the islands. This would result in a decreasing indium composition and a constant antimony composition. The possibility would suggest that the islands are actually InTlSb.

2)   The thallium is not incorporating into the layers and there is in fact elemental thallium on the surface. This result would show a decrease in the composition of both indium and

antimony. This possibility would suggest either thallium coated InSb islands or elemental thallium islands.

Since the slope of the antimony curve is less than that of the indium curve, it is possible that both of these reactions are occurring simultaneously. The result is that the islands could be composed of InTlSb and/or InSb coated with thallium. The data shows that the thallium island size is directly related to the substrate temperature. Moreover, it is a strong possibility that the islands are InTlSb. A slight reduction in the bandgap has been observed from some of these samples, Table 10, but it is not clear what is the physical origin. Further work is required to more fully understand thallium incorporation in InSb.

Table 10.   Preliminary data obtained for the growth of InTlSb by MBE

Sample/Block	Temperature	Thallium	FIR/PC (μm)
SSMBE016/(3)	T_t-40 (394)	490/420	5.9
SSMBE017/(2)	T_t-100 (360)	430	5.6
SSMBE018/(3)	T_t-20 (420)	440	6.0
SSMBE019/(2)	T_t-100 (360)	440	6.3
SSMBE021/(2)	T_t+4 (464)	440	5.7
SSMBE024/(2)	T_t-180 (280)	430	6.8
SSMBE024/(3)	T_t-240 (210)	460	5.6
SSMBE026/(2)	T_t+4 (480)	-	5.6

5.   RECENT PUBLICATIONS

MBE Growth of High Quality InSb Optimized Using RHEED Oscillations
E. Michel, G. Singh, S. Slivken, C. Besikci, P. Bove, I. Ferguson, and M. Razeghi
Applied Physics Letters

Molecular Beam Epitaxial Growth of High Quality InSb for p-i-n Photodetectors
G. Singh, E. Michel, C. Jelen, S. Slivken, J. Xu, P. Bove, I. Ferguson, and M. Razeghi
Accepted for publication in Journal of Vacuum Science and technology

Molecular beam epitaxial growth of InSb p-i-n photodetectors on GaAs and Si

E. Michel, R. Peters, S. Slivken, C. Jelen, P. Bove, J. Xu, I. Ferguson and M. Razeghi

6.   SUMMARY AND CONCLUSION
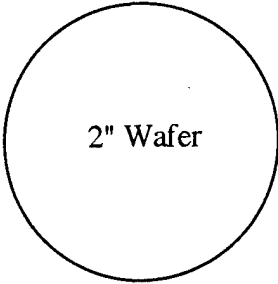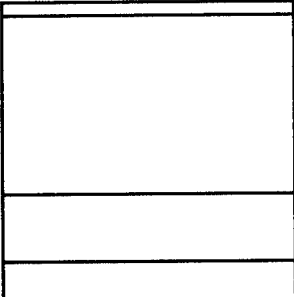
In conclusion, high quality InSb epilayers were nucleated directly onto GaAs and GaAs coated Si substrates which have exhibited the best material properties reported to date. This was achieved by accurate control of the nucleation utilizing RHEED. A 4.8μm InSb layer grown on GaAs at a growth temperature of 394 °C and a III/V incorporation ratio of 1:1.2 had an X-ray rocking curve FWHM of 148 arcsec and a Hall mobility of 92300 cm^{2}V^{-1}s^{-1} at 77K. A 4.8 μm InSb p-i-n structure exhibited a FWHM of 131 arcsec and 101 arcsec for GaAs coated Si and GaAs substrates respectively. In addition, excellent uniformity, ± 3 arcsec X-ray FWHM and morphology was obtained. A photovoltage response was obtained from prototype InSb p-i-n detectors on GaAs coated Si at 6.4 μm up to 200K with a D* of 7x10^8 cmHz$^{1/2}$W^{-1} at 77K.
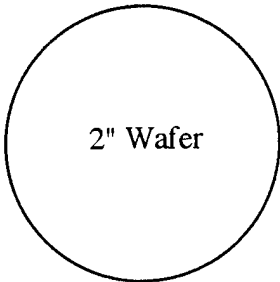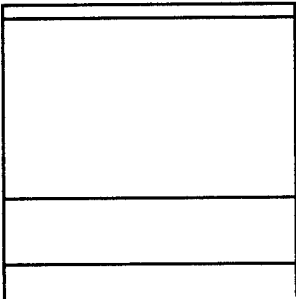
7.   REFERENCES
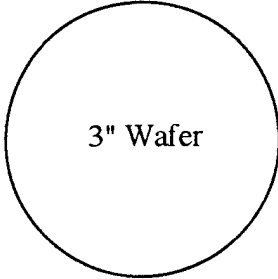
R.M. Biefield, G.A. Hebner, J. Cryst. Growth. 109, 272 (1991)

Y.H. Choi, R. Sudharsanan, C. Besikci, E. Bigan, M. Razeghi, Mat. Res. Soc. Symp. Proc. 281, 374 (1993).

J.-I. Chyi, D. Biswas, S.V. Iyer, N.S. Kumar, H. Morkoc, R. Bean, K. Zanio, H.-Y. Lee, H. Chen, Appl. Phys. Lett. 44, 1016 (1989)

J.-I. Chyi, S. Kalem, N.S. Kumar, C.W. Litton, H. Morkoc, Appl. Phys. Lett. 43, 1092 (1988).

J.L. Davis, P.E. Thompson, Appl. Phys. Lett. 54, 2234 (1994).

D.K. Gaskill, G.T. Stauf, N. Bottka, Appl. Phys. Lett. 48, 1904 (1991).

G.S. Lee, P.E. Thompson, J.L. Davis, J.P. Omaggio, W.A. Schmidt, Solid-State Elec. 36, 387 (1993).

L.K. Li, Y. Hsu, W.I. Wang, J. Vac. Sci. Technol. B 11, 872 (1993).

J.R. Soderstrom, M.M. Cumming, J-Y. Yao, T.G. Anderson, Semicond. Sci. Technol. 7, 337 (1992).

P.E. Thompson, J.L. Davis , J. Waterman, R.J. Wagner, J. Appl. Phys. 42, 7166 (1991).

8. APPENDICES

8.1 Samples grown for Loral Fairchild

Sample: InSb 'pin' photodiode/Silicon	Date: June 14 1994 SSMBE: 022

Substrate Preparation:

Type: 2" GaAs Coated Silicon 4/6

Manufacturer: Army

Orientation: 2° Off

Solvent Degrease: n/a

Acid etch: n/a

Outgas: 250°C for 1 hour

Oxide Desorb: 630°C

Other: 2" Indium free block

2" Wafer

Block number: New Block (4)

See below for sample structure	Layer 1: GaAs (30mins)		Layer 2: InSb (Various see below)	
	Temperature	Comment	Temperature	Comment
Substrate	600°C	T_{od}-30°C	405°C	T_t+5°C
Silicon			1075°C	8.0e16
Beryllium			800°C	1.0e18
Gallium	985°C			
Indium			895°C	6.35e-7
Aluminum				
Thallium				
Arsenic	320°C			
Antimony			460°C	

Comments:

The GaAs buffer layer was ungrown undoped so that this sample can be used for front processing.

The estimated growth rate is 0.93 μm/hr at incorporation ratio of 1:1.2 for the indium/antimony flux ratio.

The n-type region was grown for 1hr, the undoped region for 5hr 15min and p-type region for 15 minutes.

Layer Structure:

0.23μm p-type InSb

4.9μm undoped InSb

0.93μm n-type InSb

Sample Characterization:

X-ray: FWHM 224 arcsec

Hall Effect:

CV Profile:

Ball/Stain:

PL:

PC:

Other:

Sample: InSb 'pin' photodiode/Silicon	Date: June 15 1994 SSMBE: 023

Substrate Preparation:

Type: 2" GaAs Coated Silicon 3/6

Manufacturer: Army

Orientation: 2° Off

Solvent Degrease: n/a

Acid etch: n/a

Outgas: 250°C for 1 hour

Oxide Desorb: 630°C

Other: 2" Indium free block

2" Wafer

Block number: New Block (1)

See below for sample structure	Layer 1: GaAs (30mins)		Layer 2: InSb (Various see below)	
	Temperature	Comment	Temperature	Comment
Substrate	590°C	T_{od}-40°C	Various	T_t+5°C
Silicon			1075°C	p: 8.0e16
Beryllium			800°C	n: 1.0e18
Gallium	990°C			
Indium			895°C	BEP: 6.4e-7
Aluminum				
Thallium				
Arsenic	310°C			
Antimony			463°C	

Comments:

The GaAs buffer layer was grown n-type doped so that this sample can be used for rear processing.

The estimated growth rate is 0.93 μm/hr at incorporation ratio of 1:1.2 for the indium/antimony flux ratio.

The n-type region was grown for 33min, the undoped region for 7hr 45min and p-type region for 15 minutes.

Doping levels are nominal.   Antimony at 465°C   BEP: 2.9e-6

Sample temperature decreased as T_t changed during growth. 415°C to 405°C (1'15")to 395°C (4'45")

Layer Structure:

0.2μm p-type InSb

7.2μm undoped InSb

0.5μm n-type InSb

Sample Characterization:

X-ray: 334 arcsec

Hall Effect:

CV Profile:

Ball/Stain:

PL:

PC:

Other:

Sample: InSb 'pin' photodiode/Silicon	Date: Aug. 19, 1994 SSMBE: 039

Substrate Preparation:

Type: 3" GaAs/Si

Manufacturer: Kopin 24436

Orientation:

Solvent Degrease: n/a

Acid etch: n/a

Outgas: 250°C for 1 hour

Oxide Desorb: 600°C

Other: 3" Indium free block

3" Wafer

Block number: New Block (4)
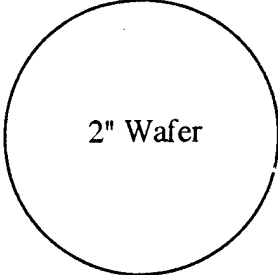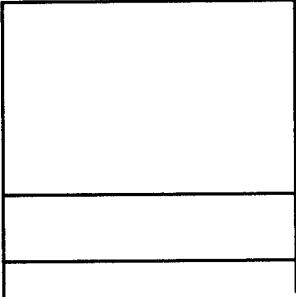
See below for sample structure	Layer 1: GaAs (1hr 10min)		Layer 2: InSb (Various see below)	
	Temperature	Comment	Temperature	Comment
Substrate	560°C	T_{od}-40°C	400°C	
Silicon			1075°C	8.0e16
Beryllium			820°C	1.0e18
Gallium	925°C			
Indium			850°C	
Aluminum				
Thallium				
Arsenic	330°C			
Antimony			415°C	
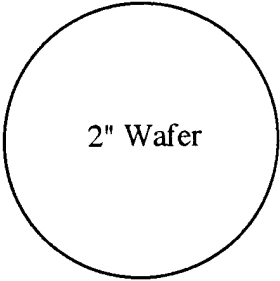
Comments:
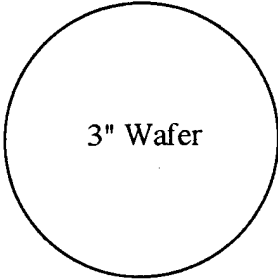
No apparent defects during GaAs buffer layer, but possible shadow from cleaning.
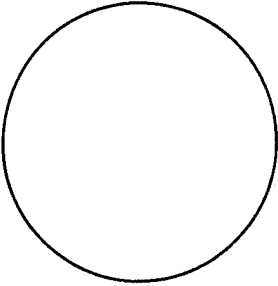
Growth stopped about one hour into undoped region due to defects.
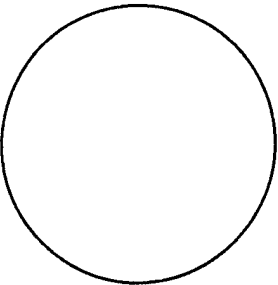
Layer Structure:

≈ 1 μm undoped InSb

1.0 μm n-type InSb

Sample Characterization:

X-ray: FWHM 224 arcsec

Hall Effect:

CV Profile:

Ball/Stain:

PL:

PC:
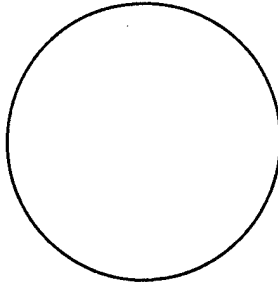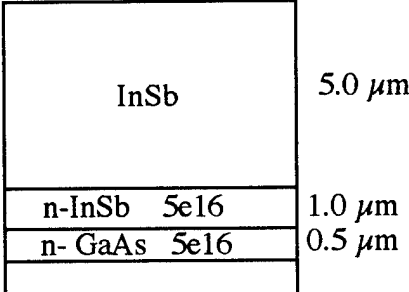
Other:

Sample: InSb 'pin' photodiode/Silicon	Date: Aug. 24, 1994 SSMBE: 041

Substrate Preparation:

Type: 2" GaAs Coated Silicon 4/6

Manufacturer: Army

Orientation: 2° Off

Solvent Degrease: n/a

Acid etch: n/a

Outgas: 225°C for 2 hours

Oxide Desorb: 630°C

Other: 2" Indium free block

2" Wafer

Block number: 2" Indium free

See below for sample structure	Layer 1: GaAs (1hr)		Layer 2: InSb (Various see below)	
	Temperature	Comment	Temperature	Comment
Substrate	550°C	T_{od}-50°C	405°C	
Silicon			1075°C	8.0e16
Beryllium				
Gallium	990°C			
Indium			850°C	
Aluminum				
Thallium				
Arsenic	315°C			
Antimony			417°C	

Comments:

LAYER MELTED

The estimated growth rate is 0.93 μm/hr at incorporation ratio of 1:1.2 for the indium/antimony flux ratio.

The n-type region was grown for 1.5 hrs., the undoped region for 5 hr 23 min.

Layer Structure:

5.0 μm undoped InSb

1.4 μm n-type InSb

Sample Characterization:

X-ray:

Hall Effect:

CV Profile:

Ball/Stain:

PL:

PC:
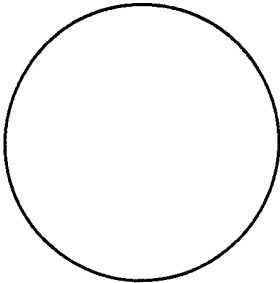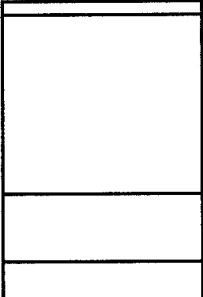
Other:

Sample: InSb 'pin' photodiode/Silicon	Date: Aug. 26, 1994 SSMBE: 043

Substrate Preparation:

Type: 2" GaAs Coated Silicon

Manufacturer:

Orientation:

Solvent Degrease: n/a

Acid etch: n/a

Outgas: 225°C for 2 hours

Oxide Desorb: 640°C

Other:

2" Wafer

Block number: 2" Indium free (4)

See below for sample structure	Layer 1: Si doped InSb		Layer 2: undoped InSb	
	Temperature	Comment	Temperature	Comment
Substrate	400°C		400°C	
Silicon	1075°C			
Beryllium				
Gallium				
Indium	850°C		850°C	
Aluminum				
Thallium				
Arsenic				
Antimony	417°C		417°C	

Comments:
The GaAs coated substrate had a cleaning/growth defect before it was loaded into the growth system.

The gallium cell is not operable therefore the oxide was desorbed with T_{As} @ 330°C and and then n-type InSb was nucleated. The X-ray measurements indicate that this has not effected the crystalline quality. The estimated growth rate is 0.93 μm/hr at incorporation ratio of 1:1.2 for the indium/antimony flux ratio.

No p-type cap growth on this layer.

Layer Structure:

5.5 μm u/d InSb
1.1 μm n-type InSb

Sample Characterization:

X-ray: 180 arcsec

Hall Effect:

CV Profile:

Ball/Stain:

PL:

PC:

Other:
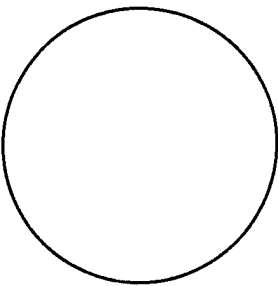
Sample: InSb 'pin' photodiode/Silicon	Date: Aug. 27, 1994 SSMBE: 044

Substrate Preparation:

Type: 2" GaAs Coated Silicon

Manufacturer: Kopin

Orientation: 2° or 4°

Solvent Degrease: n/a

Acid etch: n/a

Outgas: 225°C for 2 hours

Oxide Desorb: 630°C (10 minutes at 650°C)

Other:

3" Wafer

Block number: Epi 3" Indium free

See below for sample structure	Layer 1: Si doped InSb (1h 5min)		Layer 2: undoped InSb (6h 7min)	
	Temperature	Comment	Temperature	Comment
Substrate	410-430°C	410°C/20min	430°C	
Silicon	1075°C			
Beryllium				
Gallium				
Indium	851°C		851°C	
Aluminum				
Thallium				
Arsenic				
Antimony	418°C		418°C	

Comments:

The is on a new EPI block that has a PBN backing plate.

The gallium cell is not operable therefore the oxide was desorbed with T_{As} @ 330°C and and then n-type InSb was nucleated.

Sample becomes milky towards the sample edge and X-ray FWHM increases to ~350arcsec.

No p-type cap growth on this layer.

Layer Structure:

6.0 μm u/d InSb
1.0 μm n-type InSb

Sample Characterization:

X-ray: 160 arcsec

Hall Effect:

CV Profile:

Ball/step: 7 μm (Total thickness)

PL:

PC:

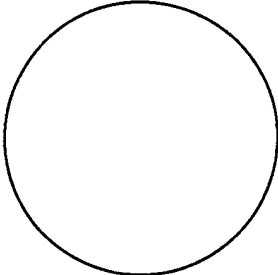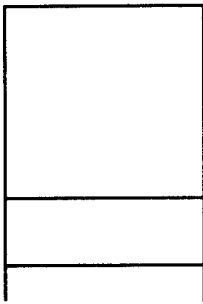Other:

Sample: 2" InSb i-n structure	Date: 9/16/94	SSMBE: 049

Substrate Preparation:

Type: semi-insulating GaAs

Manufacturer: Sumitomo

Orientation:

Solvent Degrease:

Acid etch:

Outgas: 1 hr @ 225 °C

Oxide Desorb: 640 °C

Other:

Block number: 2" block

See below for sample structure	Layer 1: n-InSb Time: 1 hr 5 min		Layer 2: InSb Time: 5 hr 20 min	
	Temperature	Comment	Temperature	Comment
Substrate	410		410	
Silicon	1075	$\approx 5 \times 10^{16}$		
Beryllium				
Gallium				
Indium	935	0.88 ML/sec	935	0.88 ML/sec
Aluminum				
Thallium				
Arsenic				
Antimony	425		425	

Comments: Undoped GaAs buffer layer grown for 32 min prior to InSb growth.
$T_{Ga} = 1125$, $T_{As} = 335$, $T_{sub} = 600$

Layer Structure:

5 μm InSb

1 μm n-InSb

GaAs buffer

S-I GaAs substrate

Sample Characterization:

X-ray: 101-105 arcsec

Hall Effect:

CV Profile:

Ball/Stain:

PL:

PC:

Other:

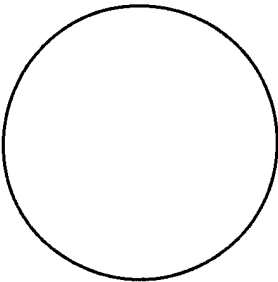| Sample: 3" InSb 'i-n'	Date: 9/26/94	SSMBE: 052

Substrate Preparation:

<u>Type</u>: GaAs coated Si

<u>Manufacturer</u>: Kopin

<u>Orientation</u>:

<u>Solvent Degrease</u>:

<u>Acid etch</u>:

<u>Outgas</u>: 1hr @ 225 °C

<u>Oxide Desorb</u>: $T_{OD} = 720$ °C

<u>Other</u>:

Block number: 3" PBN

See below for sample structure	Layer 1: n-InSb Time:1hr 5min		Layer 2: InSb Time: 5hr 40min	
	Temperature	Comment	Temperature	Comment
Substrate	435		435	
Silicon	1075			
Beryllium				
Gallium				
Indium	927		927	
Aluminum				
Thallium				
Arsenic				
Antimony	427		427	

Comments:

$n\text{-GaAs} : T_{Ga} = 1125 \text{ °C}, T_{As} = 335 \text{ °C}, T_{sub} = 690, \text{ for 30 min}$

Layer Structure:

InSb	5.0 μm
n-InSb 5e16	1.0 μm
n- GaAs 5e16	0.5 μm

Sample Characterization:

<u>X-ray</u>: 147 arcsec

<u>Hall Effect</u>:

<u>CV Profile</u>:

<u>Ball/Stain</u>:

<u>PL</u>:

<u>PC</u>:

<u>Other</u>: alphastep - 5.74 μm

Sample: 3" InSb 'i-n'	Date: 9/26/94		SSMBE: 053

Substrate Preparation:

Type: GaAs coated Si

Manufacturer: Kopin

Orientation:

Solvent Degrease:

Acid etch:

Outgas: 1hr @ 225 °C

Oxide Desorb: T_{OD} = 700 °C

Other:

Block number: 3" PBN

See below for sample structure	Layer 1: n-InSb	Time:1hr 11min	Layer 2: InSb	Time: 7hr 5min
	Temperature	Comment	Temperature	Comment
Substrate	415		415	
Silicon	1075			
Beryllium				
Gallium				
Indium	928		928	
Aluminum				
Thallium				
Arsenic				
Antimony	427		427	

Comments:

n-GaAs : T_{Ga} = 1125 °C, T_{As} = 335 °C, T_{sub} = 660, for 30 min

T_T = 410 °C

Layer Structure:

InSb	5.0 μm
n-InSb 5e16	1.0 μm
n- GaAs 5e16	0.5 μm

Sample Characterization:

X-ray: 147 arcsec

Hall Effect:

CV Profile:

Ball/Stain:

PL:

PC:

Other: alphastep - 5.74 μm

Sample: InSb p-i(π)-n	Date: 1/10/95 SSMBE: 136

Substrate Preparation:

Type: GaAs coated Si

Manufacturer: Kopin

Orientation:

Solvent Degrease:

Acid etch:

Outgas:

Oxide Desorb: $T_{od} = 700$

Other:

Block number: 3" PBN

See below for sample structure	# 1: n-InSb Time: I hr		# 2: π-InSb Time: 5 hr		# 3: p-InSb Time: 30 min	
	Temperature	Comment	Temperature	Comment	Temperature	Comment
Substrate	460		440		440	
GaTe	475	1e17				
Beryllium			700		875	1e18
Gallium						
Indium	920	0.97 μm/hr	920		920	
Aluminum						
Antimony 2						
Arsenic						
Antimony 1		407	407		407	

Comments:

Buffer layer of GaAs (2min): $T_{sub}=660$, $T_{Ga}=1084$, $T_{As}=335$ and $T_{GaTe}=475$ (1e17)

For room temperature operation

Layer Structure:

0.5μm p-type (5e18) InSb

5μm π-type (2e16) InSb

0.5μm n-type (1e17) InSb

Sample Characterization:

X-ray:

Hall Effect:

CV Profile:

Ball/Stain:

PL:

PC:

Other:

| Sample: InSb i-n structure | Date: 1/11/95 SSMBE: 137 |

Substrate Preparation:

Type: GaAs coated Si

Manufacturer: Kopin

Orientation:

Solvent Degrease:

Acid etch:

Outgas:

Oxide Desorb: $T_{od} = 710$

Other:

Block number: 3" PBN

See below for sample structure	# 1: GaAs Time: 2 min		# 2: n-InSb Time: 1 hour		# 3: InSb Time: 6 hours	
	Temperature	Comment	Temperature	Comment	Temperature	Comment
Substrate	670	Tod-40	450		450	
GaTe	450	5e16	450	5e16		
Beryllium						
Gallium	1084					
Indium			920		920	
Aluminum						
Antimony 2						
Arsenic	335					
Antimony 1		407	407		407	

Comments:

No p-type layer as this is ion implanted.

Layer Structure:

6 μm undoped InSb

1 μm n-type (5e16) InSb

Sample Characterization:

X-ray: 148 arcsec

Hall Effect:

CV Profile:

Ball/Stain:

PL:

PC:

Other:

Sample: 3" InSb i-n structure			Date: 1/12/95		SSMBE: 138	

Substrate Preparation:

Type: 3" GaAs coated Si

Manufacturer: Kopin

Orientation:

Solvent Degrease:

Acid etch:

Outgas:

Oxide Desorb: $T_{od} = 720$

Other:

Block number: 3" PBN

See below for sample structure	# 1: GaAs Time: 2 min		# 2: n-InSb Time: 1' 20"		# 3: InSb Time: 6' 11"	
	Temperature	Comment	Temperature	Comment	Temperature	Comment
Substrate	680	Tod-40	460	Tt +10	425	
GaTe	450	5e16	450	5e16		
Beryllium						
Gallium	1084					
Indium			920		920	
Aluminum						
Antimony 2						
Arsenic	335					
Antimony 1		407	407		407	

Comments:

T_t @ 450 °C. No p-type layer as this is ion implanted.

Layer Structure:

6 μm undoped InSb

1 μm n-type (5e16) InSb

Sample Characterization:

X-ray: 153 arcsec

Hall Effect:

CV Profile:

Ball/Stain:

PL:

PC:

Other:

Sample: 3" InSb i-n structure	Date: 1/16/95 SSMBE: 142

Substrate Preparation:

Type: 3" GaAs coated Si

Manufacturer: Kopin

Orientation:

Solvent Degrease:

Acid etch:

Outgas:

Oxide Desorb: $T_{od} = 720$

Other:

Block number: 3" PBN

See below for sample structure	#1: GaAs Time: 3 min		#2: n-InSb Time: 1' 20"		#3: InSb Time: 6' 11"	
	Temperature	Comment	Temperature	Comment	Temperature	Comment
Substrate	680	Tod-40	450		450	
GaTe	450	5e16	450	5e16		
Beryllium						
Gallium	1084					
Indium			920		920	
Aluminum						
Antimony 2						
Arsenic	325					
Antimony 1		407	407		407	

Comments:

Layer Structure:

6 μm undoped InSb

1 μm n-type (5e16) InSb

Sample Characterization:

X-ray: 153 arcsec

Hall Effect:

CV Profile:

Ball/Stain:

PL:

PC:

Other:

8.2 Letter from Loral Fairchild discussing InSb material quality